

12-13-89



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
WASHINGTON, D.C. 20460

DEC 13 1989

**MEMORANDUM**OFFICE OF  
PESTICIDES AND TOXIC SUBSTANCES

SUBJECT: EPA Reg No. 1471-70. Elanco trifluralin nitrosamine data of 8/18/89 in response to Data Call-In Notice under FIFRA Section 3(c)2(B). No MRID No. DEB No. 5775.

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TO: Joanne I. Miller / Mary C. Erumsele, PM Team 23  
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Elanco has submitted in response to the trifluralin DCI Notice under FIFRA, Section 3(c)2(B), summary nitrosamine data for: (1) several **Treflan**® granular products produced and sold in US from 4/89 through 8/89, and (2) a trifluralin technical and 3 liquid **Treflan**® formulations manufactured from 2/89 through 8/89. Also provided was an incomplete quality control procedure dated 8/19/82. "All campaigns were monitored for N-nitrosamines as outlined by the 'Military Standard Sampling Procedure: Military Standard-414', 11, June, 1957 with a variable sampling procedure." The "Gas Chromatography - Thermal Energy Analyzer (analytical) method" description and "products specifications" were not attached as indicated. We require them along with the raw data to verify the summary results depicted below.

**"Total Nitrosamines PPM"**

<u>Granular product</u>	<u>Contract Mfg</u>	<u>Prodn time</u>	<u>Lots</u>	<u>range ppm</u>
ID5535, Treflan TR10	Trekker Chml.	Jan/May 89	40	0 - 0.07
" " "	"	Jun/Jul 89	25	0 - 0.09
ID5901, Treflan 5G	"	Jul 89	25	0 - 0.04
ID5535, Treflan TR10	Farmland Ind.	Jun/Aug 89	35	0 - 0.04

"Nitrosamine (PPM)"

<u>Liquid frmln</u>	<u>Prodn period</u>	<u>Lots</u>	<u>range ppm</u>
Treflan E.C. ID5011	3/30/89-5/24/89	19	0.01 - 0.27
Treflan 5 ID5555	3/1/89-7/26/89	97	0 - 0.24
Treflan MTF ID5560	3/21/89-5/15/89	19	0 - 0.14
Trifluralin Tech QA114Z	2/89-6/89	404	0 - 0.47

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The current, 8/19/82, QC procedure description titled, "Quality control procedure for assuring products formulated with trifluralin meet the requirements as published in the federal register notice of August 4, 1982 (47. FED. REG. 33777-33784)", though sans attachments (i.e., Analytical method, Product specifications [CSF?]), states (in part):

"The N-nitrosamine content of trifluralin technical must be no more than 0.5 ppm.

"Appropriate sampling and testing will be done for to assure that the formulation process does not increase the N-nitrosamine content of the final third party formulated products to violative levels.

"The number of lots sampled will be determined by the aforesaid Mil-Std-414, with variation. The acceptance procedure will be based on a 5% acceptance probability at a lot tolerance percent defective of 10%.

"Data to be analyzed to determine if there has been a significant increase in N-nitrosamine content due to the formulation process.

Conclusions and Recommendation:

The summary nitrosamine assay results are **not** verifiable.

The quality control procedure cites attachments (analytical method description and product specifications), which were not provided. Also, this QC procedure description is not sufficiently specific, in regard to product-specific formulation parameters.

We recommend that Elanco, at a minimum, do the following:

1. Submit a detailed description of the analytical method, raw data (calibration curve, peak heights, concentrations & identities of standards), chromatograms for spiked samples,

spiking level, linearity of concentration/detector response ratios, recoveries, precision, background levels, and sample calculations.

2. Submit recovery data on all products to show that the method measures the trifluralin theoretical contaminants N-nitrosodipropylamine (NDPA) and C7/C8 nitrosamines. For the latter we suggest spiking the samples with nitrosopropylbutylamine and nitrosodibutylamine. We suggest spiking levels of  $\leq 0.5$  ppm for NDPA, and  $\leq 0.1$  ppm for the other two compounds.

3. Submit chromatograms and raw data from duplicate analyses (e.g., peak areas, sample weights, volumes, calculations) so that we can verify the reported recoveries and nitrosamine concentrations. The actual equations used for calculations should be clearly specified. It should also be distinctly indicated whether the samples are from two separate production batches or merely replicates of one batch.

4. Additionally, data may be required if it is shown that the method would not detect the presence of the less volatile C7-C8 nitrosamines.

5. Provide a **specific** quality control procedure which includes:

a) Guidelines for controlling the heating of technical trifluralin during, e.g., storage, formulation, blending, product packaging, shipping. Data in our files documents specific procedures (i.e., maximum high temperature/time limits), developed by a registrant cooperatively with Elanco.

b) If the nitrosamine analyses were done by a contract lab, a nitrosamine monitoring plan which addresses how these contaminants will be monitored in each batch of product prior to shipping.

c) Certification that no nitrite is used in the packaging material.

6. Provide current Confidential Statements of Formula for all products.

**PM NOTE:** We suggest that Elanco be sent a complete copy of this review.

cc: K. Dockter (DEB), D. Schmitt, Trifluralin & Nitrosamine SFs, E. Eldredge (ISB/PMSD), Circulation (7), RF.

RDI: AARathman:12/11/89:EZager:12/11/89

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